

PATENT SPECIFICATION

NO DRAWINGS

L120.309



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Date of Application and filing Complete Specification: 26 April, 1966.
No. 18272/66.

Application made in United States of America (No. 451021) on 26 April, 1965.
Complete Specification Published: 17 July, 1968.

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Index at acceptance:—C2 C(2A2, 2A11, 2R15, 2T16, 3A7V2A2, 3A7V2L, 3A7V2Q, 3A7V4A2, 3A7V4E1, 3A7V4L, 3A10A4E, 3A10A4F, 3A10A5K, 3A10B2C, 3A10B5K)

Int. Cl.:—C 07 c 63/52, C 07 c 65/02, C 07 c 87/28, C 07 c 121/66

COMPLETE SPECIFICATION

Process for Arylating Unsaturated Fatty Compounds

We, ARMOUR AND COMPANY a corporation organised and existing under the laws of the State of Delaware, United States of America of 401 North Wabash Avenue, Chicago, Illinois 60611, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to a process for arylating unsaturated fatty compounds, such as unsaturated fatty acids and their derivatives.

It has previously been proposed to arylate unsaturated fatty compounds, such as oleic acid, with arylating agents such as benzene, toluene, naphthalene, and other aromatic hydrocarbons employing liquid hydrofluoric acid as the catalytic reaction medium. As illustrative of such prior art, reference may be made to U.S. Patent 2,275,312 and J. Am. Chem. Soc. 61, 1010—1015 (1939). As far as is known, however, no process of this kind has gone into commercial use, probably because of the relatively poor yields reported in the literature. While in some cases yields as high as 50—60% have been obtained, for commercial applications much higher yields are desirable, up to 90% or better. Previously, no improvements have been proposed which are capable of improving yields, and which adapt the reaction of those compounds with arylating agents in hydrofluoric acid for commercial use, either as a batch or a continuous process.

It is, therefore, an object of the present invention to develop an improved process for arylating unsaturated fatty compounds in hydrofluoric acid whereby good yields can be obtained in a reasonable time, and which can be operated as a batch or continuous process.

[Price 4s. 6d.]

Accordingly the invention provides a process for arylating unsaturated fatty compounds having an unsaturated aliphatic hydrocarbon group of from 8 to 22 carbon atoms, but having no other groups which are susceptible to arylation under the conditions of the reaction, comprising the step of reacting one mole of the fatty compound with at least 2 moles of arylating agent in the presence of at least 2 moles of liquid hydrofluoric acid and allowing the reaction to continue until substantially all of the fatty compound has reacted.

In this specification arylation is taken to mean the formation of a new carbon to carbon bond between the fatty compound and the aromatic nucleus.

The process of this invention is carried out in liquid hydrofluoric acid. If the reaction is to be conducted at temperatures above the boiling point of hydrofluoric acid, super-atmospheric pressure may be used to maintain the hydrofluoric acid as a liquid. As long as the hydrofluoric acid is maintained in the liquid state, it functions both as a catalyst for the reaction and as the reaction medium or solvent. Consequently, it is possible to carry out the reaction over a broad range of temperatures, viz., -20° C. to 100° C. Usually, however, it is preferable to conduct the reaction at a temperature within the range from 0° to 25° C.

Virtually any aromatic hydrocarbon may be used as the arylating agent. Arylating agents having a benzene nucleus are preferred, but other arylating agents can be used, such as naphthalene, anthracene, phenanthrene, etc. Generally speaking, the arylating agent should have an aromatic hydrocarbon nucleus, which may be either substituted or unsubstituted. Among the preferred arylating agents are benzene, toluene, xylene, anisole, cresol,

phenol, and resorcinol. A preferred sub-class of arylating agents includes benzene or benzene substituted with 1 or 2 methyl, methoxyl, or hydroxyl groups.

5 The unsaturated fatty compound is preferably a long chain unsaturated fatty acid or unsaturated derivative thereof. More generally, the present process is applicable to unsaturated fatty or aliphatic compounds having an
10 aliphatic hydrocarbon chain of from 8 to 22 carbon atoms with at least one unsaturated or olefinic linkage. Unsaturated fatty acids obtained from animal or vegetable fats and oils are particularly suitable, as are their
15 derivatives, such as fatty esters (e.g., triglycerides), fatty amines and fatty nitriles. Specific preferred examples of fatty acid compounds which may be used are oleic acid, 9-octadecenyl amine, erucic acid, unhydrogenated
20 tallow, and castor oil. Other examples include linoleic acid, ricinoleic acid, and 10-undecenoic acid. Mixtures of different unsaturated fatty compounds can be used, and other saturated fatty compounds which will not react
25 may also be present. Particularly good results have been obtained with oleic acid and derivatives thereof such as 9-octadecenyl amine and nitrile.

30 The arylation of 9-octadecenyl nitrile presents a particular problem, since the nitrile group can react with the olefinic linkage. The process of this invention, however, promotes the desired arylation reaction while minimizing the undesired reaction between the nitrile
35 groups and olefin groups. The previous processes have not been adapted to the arylation of 9-octadecenyl nitrile, relatively low yields being obtained even when the reactant was oleic acid.

40 When an unsaturated fatty acid is arylated with phenol, some esterification may occur as a side reaction between the hydroxyl and carbonyl groups. This side reaction, however, is not unduly detrimental to the main arylation
45 reaction which proceeds unaffected. Any portion of the product in the ester form can be easily hydrolyzed to the free acid, if desired.

50 The present invention is based in part on the discovery that the process can be greatly improved by employing a large molar excess of the arylating agent. As will subsequently be explained in detail, this procedure has been found to increase greatly the yield of the
55 arylated derivative, and also apparently reduces the required reaction time, making it feasible to adapt the process for continuous operation. It has also been discovered that the excess arylating agent need not be wasted,
60 but can readily be recovered and recycled through the process.

65 In accordance with the present invention, at least 2 moles of the arylating agent should be employed per mole of the unsaturated fatty compound. Optimum results are obtained

within the range from 2.5 to 6 moles of the arylating agent per mole of the unsaturated fatty compound. A larger excess than 6 moles can be employed, but there is no particular advantage. In some cases, however, it
70 might be feasible to use up to 10 moles of the arylating agent per mole of the unsaturated fatty compound.

75 It has also been found that the use of an insufficient amount of hydrofluoric acid tends to reduce the yield. As a minimum, at least 2 moles of hydrofluoric acid should be employed per mole of the unsaturated fatty compound, and preferably at least 3 moles of
80 hydrofluoric acid per mole of the compound. The preferred range is from 3 to 15 moles of hydrofluoric acid per mole of unsaturated fatty compound, although a greater ratio of hydrofluoric acid can be used, up to 30 moles
85 per mole of unsaturated fatty compound. However, it has been found that there is no particular advantage to employing more than 15 moles of hydrofluoric acid per mole of unsaturated fatty compound.

90 When the process of the present invention is conducted in the preferred manner, the desired reaction proceeds very rapidly. Usually it is not necessary or desirable to continue the reaction for more than four hours, and in many cases a much shorter time can be used. Under
95 optimum conditions, the reaction proceeds to completion in less than one hour after the reactants have been introduced into the hydrofluoric acid. For example, reaction times of 5 to 30 minutes may be used, thereby making
100 the process readily adaptable to continuous operation.

105 The reactants may be introduced separately or simultaneously into the hydrofluoric acid. For batch reactions, it will usually be most convenient to introduce first the arylating agent, thereby making sure that a large excess of the arylating agent is present. Thereafter,
110 the oleic acid or other unsaturated fatty compound can be introduced gradually while stirring the reaction mixture.

115 After the completion of the reaction, the arylated compound may be recovered in various ways. For instance, the reaction mixture can be added to water, and the arylated compound extracted from the water into an organic solvent, such as diethyl ether or benzene. The ether and any excess of the arylating agent can be stripped off by vacuum
120 distillation, leaving the arylated compound.

125 In a preferred embodiment of the present invention, the hydrofluoric acid and the excess arylating agent are removed from the arylated compound upon the completion of the reaction by distillation. The crude reaction product which is left as a residue can be further purified, if desired, by distillation. When the excess arylating agent and hydrofluoric acid are removed together by distillation, they can be condensed, and returned
130

to the process. This procedure is particularly desirable where a large excess of the arylating agent is employed, as described above.

- 5 This invention is further illustrated by the following examples:

EXAMPLE 1

10 A 5-liter polyethylene beaker, equipped with a mechanical stirrer and thermocouple, was charged with 616 g. (30.8 moles) of liquid, anhydrous hydrogen fluoride at 0 to -5° C. About 300 g. (3.26 moles) of toluene were added to the hydrogen fluoride. A solution of oleic acid (800 g., 2.83 moles) in 850 g. (9.25 moles) toluene was then added to the reaction mixture at $5-15^{\circ}$ C. over a 2-3 hour period with vigorous stirring. Stirring was continued for an additional hour; then the reaction mixture was poured into 8 liters of cold water. Four liters of ethyl ether were added and the organic layer separated and washed with one-liter portions of water until the washings were neutral. The ether and excess toluene were stripped off *in vacuo* to yield 1017.4 g. (96.0% of theory) of tolylstearic acid as a viscous amber oil.

Analysis:

Neut. equivalent—365.0 (calcd. 374.6)

Iodine value 3.59

30 Molecular distillation of the crude acid at $167-188^{\circ}\text{C}/165-190\mu$ afforded a 68% yield of pure tolylstearic acid. A fraction boiling at $199-251^{\circ}\text{C.}/0.5-7\mu$, amounting to 10-12%, was also isolated corresponding to methylphenylene distearic acid.

EXAMPLE 2

40 A 5-liter polyethylene beaker, equipped with a mechanical stirrer and thermocouple, was charged with 826 g. (41.3 moles) of liquid, anhydrous hydrogen fluoride at 0 to -5° C. About 200 g. (2.17 moles) of toluene was added to the hydrogen fluoride. A solution of tallow acid (929 g., 3.36 moles) in toluene (595 g., 6.45 moles) was then added to the reaction mixture at $5-15^{\circ}$ C. over a period of 1-8 hours with vigorous stirring. Stirring was continued for one hour; then the reaction mixture was poured into 8 liters of cold water. Four liters of ether were added, the organic layer separated and washed with one-liter portions of water until the washings were neutral. The ether and excess toluene were stripped off *in vacuo* to yield 1060.7 g. of crude tolyltallow acid as a solid.

55 Analysis:

Neutralization equivalent—317.6 (Calcd 332.5)

Iodine value — 4.15

EXAMPLE 3

60 A two-liter polypropylene beaker equipped with a magnetic stirrer and thermocouple was

charged with 400 g. (20 moles) liquid, anhydrous hydrogen fluoride and 220 ml. (192 g., 2.47 moles) of benzene. A solution of commercial 9-octadecenyl nitrile (427 g., 1.62 moles) in benzene (500 ml., 440 g., 5.63 moles) was added over a one-hour period with stirring at $0-10^{\circ}$ C. The reaction mixture was stirred for an additional 45 minutes at $5-10^{\circ}$ C., then poured into 8 liters of cold water. Ether (1.5-2 liters) was added and the aqueous layer separated. The organic layer was washed seven times with 300 ml. portions of water until the washings were neutral. The ether solution was then dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 508.7 g. (92% mass yield) of an amber oil.

Analysis:

21% Monosubstituted amide (Infrared analysis)

Gas chromatographic analysis (corrected for amide)

67% phenylstearonitrile

11.9% saturated starting nitriles

Molecular distillation of the crude nitrile at $138-153^{\circ}\text{C.}/47-75\mu$ afforded a 58.5% yield of pure phenylstearonitrile.

EXAMPLE 4

A one-liter Monel (Registered Trade Mark) reactor was charged with 96 g. (4.8 moles) of liquid anhydrous hydrogen fluoride and 265 g. (2.88 moles) of toluene at $0-5^{\circ}$ C. The reactor was sealed and the temperature of the reaction mixture was raised to 21° C. A solution of 270 g. (0.96 mole) commercial grade of oleic acid in 88 g. (0.957 mole) of toluene was pumped into the toluene-hydrogen fluoride mixture at a constant temperature of 21° C. over a period of 90 minutes. The reaction mixture was stirred for an additional 30 minutes at 21° C. The reactor was fitted with a condenser and receiver and heat was applied. A continuous stream of N_2 was used as a carrier gas to aid in the distillation of the hydrogen fluoride and unreacted toluene. The temperature was raised gradually to 190° C., then the reaction mixture was cooled to yield 342 g. (97.2% mass yield) of a dark oil.

Analysis:

Neutralization equivalent, 373 (calcd 374.6)

Iodine value, 2.7

% HF, trace

Distillation of the crude product resulted in three fractions:

Light ends 3.25%

Main fraction 83.5 %

Residue 11.0 %

Distillation losses 2.25%

Analysis of the main fraction by GLPC showed:

Unreacted saturated acids 3.6%

Tolylfatty acids 96.3%

Neutralization equivalent 373.

EXAMPLE 5

Additional runs were made following the procedure set out in Example 4, but varying

the mole ratio of the oleic acid, toluene, and hydrofluoric acid. The results of these tests are summarized in the following table:

TABLE A

	REACTANTS			PRODUCT		
	Mole Ratio		HF	Neut. Equivalent	Iodine Value	% Residue
	Oleic	Toluene				
10	1	1.1	3	382	8.9	33
	1	1.1	5	354	5.4	44.7
	1	2	2	369	9.3	25.2
	1	2	5	387	3.6	22.7
15	1	2	11	368	3.4	16.7
	1	2	5	387	3.6	22.7
	1	3	5	—	—	14.0
	1	4	5	373	2.7	11
20	1	5	5	374	2.6	11.3
	1	4.3	11	369	4.1	10

In the above table, the percent residue varies inversely with the degree of completion of the reaction, and hence this figure indicates the approximate yield of the desired product. The results demonstrate that a high ratio of the arylating agent to the oleic acid is essential to obtain high yields.

EXAMPLE 6

A 5-liter polyethylene beaker, equipped with a mechanical stirrer, thermocouple, and addition funnel, was charged with 520 g. (25.9 moles) of liquid, anhydrous hydrogen fluoride and 279 g. (2.62 moles) of xylene at 0—5° C. A solution of 600 g. (2.1 moles) of oleic acid in 836 g. (7.88 moles) of xylene was added with stirring over 2 hours at 10—15° C. The reaction mixture was stirred for an additional hour at 10—15° C., then poured into 6 liters of cold water. Two liters of ether were added and the aqueous phase separated. The organic layer was washed several times with salt water until the washings were neutral. The ether solution was dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 846.1 g. of a viscous amber oil, xylyl-stearic acid, in almost quantitative yield.

Analysis:

Neutralization equivalent, 399 (calc'd 388.7)
Saponification equivalent, 391
Iodine Value, 1.64

EXAMPLE 7

A 4-liter polyethylene beaker equipped with a mechanical stirrer, thermocouple and addition funnel, was charged with 456 g. (22.75 moles) of liquid, anhydrous hydrogen fluoride and 500 ml. (500 g., 4.62 moles) of anisole. A solution of oleic acid (523 g., 1.85 moles) in anisole (250 ml., 250 g., 2.3 moles) was added with stirring over a 90-minute period at 5—15° C. The reaction mixture was stirred for an additional hour

at 10—15° C., then poured into 8 liters of cold water. Ether (1.5—2 liters) was added and the aqueous phase separated. The organic layer was washed seven times with 300 ml. portions of water until the washings were neutral. The ether solution was then dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 661.5 g. (91.6% mass yield) of a yellow, low melting solid.

Analysis:

Neutralization equivalent, 385.5 (calc'd 390.6)
Infrared analysis indicated the material was the desired product.

EXAMPLE 8

A 2-liter polypropylene beaker, equipped with a mechanical stirrer, thermocouple, and addition funnel, was charged with 492 g. (24.6 moles) of liquid, anhydrous hydrogen fluoride and 82.0 g. (0.164 mole) of naphthalene. The mixture was warmed to 16° C. and a warm solution of oleic acid (137 g., 0.5 mole) and naphthalene (123 g., 0.95 mole) was added with stirring over a 45-minute period at 16—18° C. The addition funnel was then rinsed with 25 ml. of ether and the ether added to the reaction mixture. Stirring was continued for one hour at 16—18° C. after the addition was completed. The reaction mixture was then poured into 8 liters of cold water. Ether (1.5—2 liters) was added and the aqueous phase separated. The organic layer was washed several times with 300 ml. portions of water until the washings were neutral. The ether solution was then dried over anhydrous sodium sulfate and stripped under reduced pressure. Excess naphthalene was removed by sublimation *in vacuo*. Crude naphthylstearic acid was isolated as a viscous amber oil, 177.7 g. (88.4% mass yield). Analysis: Neutralization equivalent 365.5 (calc'd 402), saponification equivalent 368, Iodine Value 6.28.

EXAMPLE 9

5 A 300 ml. Monel autoclave was charged with 45 g. (2.25 moles) of liquid, anhydrous hydrogen fluoride and 66.6 ml. (59.0 g., 0.75 mole) of benzene at 0° C. The reactor was sealed and the temperature of the reaction mixture raised to 30° C. Molten erucic acid (50.8 g., 59.2 ml., 0.15 mole) was pumped into the hydrogen fluoride-benzene mixture at 25—30° C. over a 20—25 minute period. The reaction mixture was stirred for an additional 30 minutes at 25—30° C., then poured into one liter of cold water. One liter of ether was added and the aqueous phase separated. The organic layer was washed seven times with 250 ml. portions of salt water. The ether solution was dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 69.9 g. (98.9% mass yield) of a dark viscous oil.

Analysis:
Neutralization equivalent, 407 (calc'd 416.7)
Iodine value, 4.0

EXAMPLE 10

25 A 300 ml. Monel autoclave was charged with 40 g. (2.0 moles) of liquid, anhydrous hydrogen fluoride and 29 ml. (25.5 g., 0.327 mole) of benzene at 13° C. A solution of 36.9 g. (0.20 mole) of 10-undecenoic acid in 60 ml. (52.6 g., 0.673 mole) of benzene was added with stirring over a 30—35 minute period at 13—18° C. The reaction mixture was stirred for an additional 30 minutes at 14—18° C., then poured into one liter of cold water. Ether was added and the aqueous phase separated. The organic layer was washed six times with 250 ml. portions of salt water. The ether solution was dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 55.3 g. of dark, viscous oil in almost quantitative yield.

Analysis:
Neutralization equivalent, 256.5 (calc'd 262.4)

EXAMPLE 11

45 A 1000 ml. Monel autoclave was charged with 180 g. (9.0 moles) of liquid, anhydrous hydrogen fluoride and 200 ml. (176 g., 2.25 moles) of benzene at 0° C. The reactor was sealed and the temperature of the reaction mixture raised to 15° C. A solution of castor oil (280 g., 0.3 mole) in 200 ml. (176 g., 2.25 moles) of benzene was pumped into the benzene-hydrogen fluoride mixture at 15—22° C. over a 130-minute period. The reaction mixture was stirred for an additional 86 minutes at 7—20° C., then poured into 1.5—2 liters of cold water. Two liters of benzene were added and the aqueous phase separated. The organic layer was washed four times with 250 ml. portions of salt water. The benzene solution was dried over anhydrous sodium sulfate and stripped

in vacuo to yield 322.0 g. (92.0% mass yield) of a light brown, viscous oil.

Analysis:
Saponification equivalent, 373.8 (calc'd 389)
Iodine value, 13.7
Infrared analysis indicated the material to be primarily the triglyceride of phenyl hydroxystearic acid.

EXAMPLE 12

A 300 ml. Monel autoclave was charged with 56.5 g. (2.83 moles) of liquid, anhydrous hydrogen fluoride and 75.6 ml. (66.4 g., 0.85 mole) of benzene at 5° C. The reactor was sealed and the temperature of the reaction mixture raised to 14° C. Tall oil fatty acid (564 g., 0.2 mole) in 50 ml. (44 g., 0.564 mole) of benzene was pumped into the benzene-hydrogen fluoride mixture at 11—21° C. over a 50-minute period. The reaction mixture was stirred for an additional 30 minutes at 20—25° C., then poured into one liter of cold water. One liter of ether was added and the aqueous phase separated. The organic layer was washed eight times with 250 ml. portions of salt water. The ether solution was dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 68.3 g. (95% mass yield) of brown, viscous oil.

Analysis:
Neutralization equivalent, 361 (calc'd 360)
Iodine value, 30.6

EXAMPLE 13

A 300 ml. Monel stirred autoclave was charged with 40 g. (2.0 moles) of liquid, anhydrous hydrogen fluoride and 60 ml. (52.8 g., 0.68 mole) of benzene at 7° C. The reactor was sealed and a solution of 55.9 g. (0.204 mole) of 9-octadecenyl amine in 30 ml. (26.4 g., 0.34 mole) of benzene pumped into the reaction mixture at 7—16° C. over a 30—35 minute period. The reaction mixture was stirred for an additional 30 minutes at 15—22° C., then poured into 2 liters of cold water. The aqueous solution was made alkaline (pH 10) with KOH and one liter of ether added. The aqueous phase was separated and the organic layer washed four times with salt water. The ether solution was dried over anhydrous sodium sulfate and stripped *in vacuo* to yield 65.1 g. (92.5% mass yield) of phenyloctadecylamine as a yellow oil.

Analysis:
Neutralization equivalent, 334 (calc'd 345.6)
Iodine value, 4.8

EXAMPLE 14

A 300 ml. Monel stirred autoclave was charged with 40 g. (2 moles) of anhydrous, liquid hydrogen fluoride and 4.1 g. (1 mole) of phenol at 8° C. The reactor was sealed and 56.5 g. (0.2 mole) of commercial oleic

- acid was pumped into the reaction mixture at 4—8° C., over a 30—35 minute period. The reaction mixture was stirred for an additional 15 minutes at 4—6° C., then poured into 1—1.5 liters of cold water. One liter of ether was added and the aqueous phase separated. The organic layer was washed six times with 250 ml. portions of salt water and dried over anhydrous sodium sulfate. The ether solution was then stripped *in vacuo* to yield 73.3 g. (97.4% mass yield) of a light amber viscous oil.

Analysis:

- Neutralization equivalent, 463 (calc'd 376.6)
Saponification equivalent, 373

- The crude hydroxyphenylstearic acid, described above, was subjected to hydrolysis in a refluxing alcoholic solution of potassium hydroxide. The completely saponified acid was isolated as an amber viscous oil in 90.3% yield having the following analysis:
Neutralization equivalent, 350 (calc'd 376.6).

WHAT WE CLAIM IS:—

1. A process for arylating unsaturated fatty compounds having an unsaturated aliphatic hydrocarbon group of from 8 to 22 carbon atoms, but having no other groups which are susceptible to arylation under the conditions of the reaction, comprising the step of reacting one mole of the fatty compound with at least 2 moles of arylating agent in the presence of at least 2 moles of liquid hydrofluoric acid and allowing the reaction to continue until substantially all of the fatty compound has reacted.
2. A process as claimed in Claim 1 in which the unsaturated fatty compound is an acid, ester, amine or nitrile.
3. A process as claimed in Claim 1 or Claim 2 in which from 2.5 to 6 moles of arylating agent is used per mole of fatty compound.
4. A process as claimed in any of Claims 1 to 3 in which from 2 to 30 moles of liquid hydrofluoric acid is used per mole of fatty compound.

5. A process as claimed in any of Claims 1 to 4 in which from 3 to 15 moles of liquid hydrofluoric acid is used per mole of fatty compound.

6. A process as claimed in any of Claims 1 to 5 in which the excess arylating agent and hydrofluoric acid are distilled off from the product, to be recovered and re-used in the process.

7. A process as claimed in any of Claims 1 to 6 in which the fatty compound is oleic acid, 9-octadecenyl amine, erucic acid, undehydrogenated tallow or castor oil.

8. A process as claimed in any of Claims 1 to 7 in which the arylating agent is benzene, naphthalene, phenol, toluene or anisole.

9. A process as claimed in any of Claims 1 to 7 in which the arylating agent contains a benzene nucleus.

10. A process as claimed in any of Claims 1 to 7 in which the fatty compound is oleic acid and the arylating agent contains a benzene nucleus.

11. A process as claimed in any of Claims 1 to 10 in which the reaction is conducted at superatmospheric pressure.

12. A process as claimed in any of Claims 1 to 11 in which the reaction is conducted at a temperature between -20° C and 100° C.

13. A process as claimed in any of Claims 1 to 11 in which the reaction is conducted at a temperature between 0° C and 25° C.

14. A process for arylating unsaturated fatty compounds substantially as described herein with reference to any of the Examples.

15. Arylated fatty compounds when produced by any of the processes as claimed in any of Claims 1 to 14.

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